

PATENT

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re: Kendall, et al. Docket No.: IR-2795(EC)
Serial No.: 09/888,793 Filing Date: 6/25/2001
Examiner: Robert D. Harlan Art Unit: 1713
For: "Metathesis Polymerization Adhesives and Coatings"

#12
Judy
1/19/03

Dec. 12, 2002

Assistant Commissioner for Patents
Washington, DC 20231

DECLARATION PER 37 C.F.R. 1.131

Sir:

This Declaration accompanies an IDS statement submitted to the Office Action on the date noted above. A reference was cited in a foreign search report. The undersigned Applicants declare the following:

As a below named inventor, we hereby declare that:

Our residence, post office address and citizenship are as stated below next to our names; and

we believe we are the original, first and joint inventors of the subject matter which is claimed and for which a patent is sought on the invention entitled, "Metathesis Polymerization Adhesives and Coatings" the specification of which was filed 6-25-01 in the United States Patent and Trademark Office.

Submitted as attached is objective evidence of invention by Applicants prior to the reference effective date, August 10, 2000 of WO 00/46257, Applicant: Materia, Inc. as attached photocopy pages of Notebooks and is evidence of actual reduction to practice, summarized as follows:

Pages 28 and 29 from Notebook No. 8148, evidencing that polymerization between rubber and metal with bonding was achieved using Grubb's catalyst and ENB monomer.

Pages 58 and 59 from Notebook No. 8060 showing that Grubbs catalyst coatings on EPDM substrate followed by coating of p-ENB resulted in a polymerized coating; and catalyst coatings on glass slides followed by spraying of ENB monomer resulted in a peelable solid coating film.

Page 57 from Notebook No. 8148 showing that monomer mixtures of ENB and TCHP polymerized with Grubb's catalyst.

Pages 50 and 51 of Notebook No. 8297 demonstrated contact metathesis adhesion between different polyolefin (LDPE) substrate bonded to itself with 5,5-bis(chloromethyl)-2-norbornene using Grubb's catalyst.

Pages 41 and 42 of Notebook No. 8327 demonstrated the bonding of polypropylene to itself using 5,6-bis(chloromethyl)-2-norbornene delivered from a 2-part cartridge.

Pages 66 and 67 of Notebook No. 8327 demonstrated bonding of polypropylene using ENB and Grubb's catalyst.

Pages 64 to 65 of Notebook No. 8363 demonstrated formulation as a two part adhesive of ENB, NBD, silica, and an elastomer (Europrene) provided a measured amount of bond strength in lap shear samples.

Pages 11, 12, 15, 22 and 23 of Notebook No. 8374 demonstrated formulation as a two part adhesive of norbornadiene, ENB, Blendex, Cab-o-sil and Zeeospheres provided a measured bond strength in lap shear polypropylene samples.


Pages 51 to 56, 59-60 and 89-90 of Notebook No. 8374 demonstrated 2-part adhesive formulations applied to bond polypropylene substrates.

We further declare that all statements made herein of our own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States

Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Full name of first inventor:
Inventor's Signature:

Jonathan L. Kendall

 12-12-02

Date:

Dec. 12, 2002

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Full name of second inventor:
Inventor's Signature:

Kenneth C. Caster



Date:

Dec. 12, 2002

Dec. 12, 2002

Residence:

Cary, North Carolina

Citizenship:

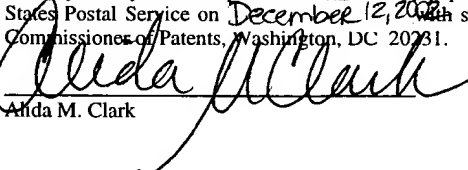
United States of America

Post Office Address:

109 Lewey Brook Drive
Cary, North Carolina 27519

CERTIFICATE OF MAILING (37 CFR 1.8(A))

I hereby certify that this paper (along with any paper referred to as being attached or enclosed) is being deposited with the United States Postal Service on December 12, 2002 with sufficient postage as first class mail in an envelope addressed to the Assistant Commissioner of Patents, Washington, DC 20231.


Aida M. Clark

Dec. 12, 2002

Date

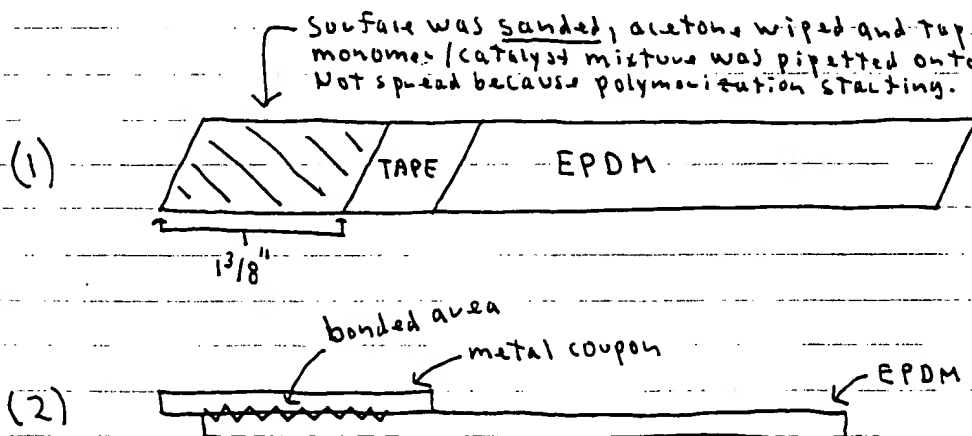
From Page No. _____

~~8148~~
Rubber to Metal Bonding Using Monomer / Catalyst Mixture

Materials: ENB (CK 8148-15-1); Grubbs's Catalyst (Strem);
EPDM (J. Taylor); Grit Blasted Metal Coupons
(M. Righettini)

Procedure:

- 1) 0.0009g of catalyst (0.000001 moles) was weighed into a glass vial.
- 2) 2.8ml (2.5004g or 0.0208 moles) of ENB was quickly syringed into the glass vial and immediately stirred for a few seconds.
- 3) The monomer / catalyst mixture was then dropped onto the EPDM strip using a glass pipet. The metal coupon was finally placed over the treated EPDM surface and held in place until bonding occurred. A brown jar was placed over the bonded area. Monomer to catalyst molar ratio was 20,800:1.
- 4) Bonding procedure was as follows (pictured from above description):



Immediately, the acetone washed metal coupon was placed on the EPDM strip in treated area. Material came out the sides and polymerized during bonding. The coupon was held in place until hard to move.

To Page No. 29

Witnessed & Understood by me,

S. E. [Signature]

Date

~~8/1/88~~

Invented by

Recorded by

Chris Keck

Date

~~8/1/88~~

From Page No. 28

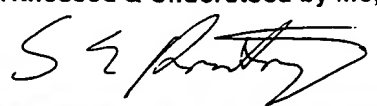
5) 5 hrs after bonding, metal easily pulled apart from rubber. Nothing on metal. Rubber had a thin, elastic film stuck to it.

PART II

Steps 1-4 (pages 28) were repeated. However, this time the monomer/catalyst mixture was pipetted onto the metal surface and the EPDM quickly placed onto the metal surface. The reverse of the first experiment. 17 hrs after bonding, metal again easily pulled apart from the rubber. Like above, nothing on metal while rubber had a thin, elastic film stuck to it.

C.K.
~~XXXXXXXXXX~~

To Page No. _____

Witnessed & Understood by me,


Date
~~XXXXXXXXXX~~

Invented by
Recorded by Chris Keck

Date
~~XXXXXXXXXX~~

From Page No. 58

Purpose: To prepare samples for surface analysis on CMP technology.
Ed Tokas will be taking different samples to NJ w/ MAS to
conduct ESCA analysis.

Instructions: from Ed Tokas

Printed By: Ed Tokas Page: 1

From: Ed Tokas (Ages 10)
To: Chris Keck, Russell Walls
CC: Ken Carter, Steve Howe, Marlene Righettini, Lynn Yanyo
REGARDING CMP Samples for Analysis
Chris and Russ,

Giving you a reminder that next week I will be going to conduct two separate surface analyses on
CMP samples:

1st Tuesday Marlene and I will go to NCSU to run a cross sectional analysis on R/R bonded
samples (one freshly prepared and one aged sample).

2nd On Wednesday I will be going to NJ w/ MAS to conduct ESCA analysis and will need the
following samples:

Labeled as
Notebook Page

Sample

- ✓ -1 EPDM
(for all preps in this series use the 1/16 in. pads
located in my office)
- ✓ -2 EPDM w/ catalyst
- 3 EPDM w/ a thin layer of poly-ENB
(thinnest of three samples)
- 4 EPDM w/ intermediate thickness of poly-ENB
- 5 EPDM w/ thick layer of poly-ENB
- 6 and -7 A free film of poly-ENB
(prepared on glass and carefully removed)
- 8 A bulk polymerized poly-ENB
(prepare in a tube)
- ✓ -9 Metal coupon
(the grit blasted Fe coupons we have been using)
- ✓ -10 Metal w/ catalyst
(the grit blasted Fe coupons we have been using)

See me Monday AM to discuss.

Thanks Ed

Russell
Walls

Procedure/Log

Time

Comments

8:30 am

Discussions with Ed Tokas to make clear what is to be done

Since the samples are to be sprayed, I was instructed to prepare
most of the samples. Chris Keck was motivated to make the
bulk polymerized poly-ENB in a small test tube.

1:00 pm

Ed Tokas wanted to be present for first spraying
0.75g of yellow catalyst was dissolved in 10ml MeCl₂ and swirled till dissolved.

1:10 pm

wiped with
acetone to
clean first.

Coated 1/16" 6"x6" EPDM with catalyst on two passes, dry, and
two more passes for a total of 4 passes. Ed said it looked
fine. Asked that we wait 30 minutes before applying monomer.

To Page No. 59

Witnessed & Understood by me,

James B. Weger

Date

~~1/11/84~~

Invent d by

Recorded by

Russell Walls

Date

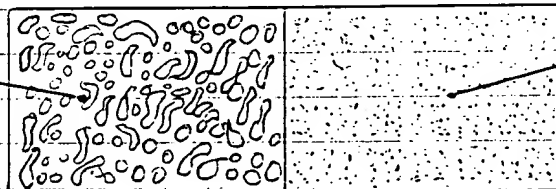
~~1/11/84~~

TITLE ESCA Samples continued

From Page No. 58

- | Time | Comments |
|---------|--|
| 1:40 pm | Applied thin coat of poly-ENB by making two passes over the rubber surface. Allowed to dry in hood. Smell of monomer still present. 1 sample with just catalyst stored. |
| 1:45 pm | Acetone wiped EPDM labelled as RW8060-58-1 with silver pen on ball and placed in ziplock bag. Bag labelled and brown paper placed in front of surface to be tested so that it would not come in contact with plastic of bag.
EPDM with just catalyst labelled as RW8060-58-2 and stored in ziplock bag just like RW8060-58-1. |
| 2:00 pm | Forgot to note that at 1:40 pm I also coated two small metal coupons (not grit blasted) with 1 ml catalyst. Stored and labelled as RW8060-58-10 and placed in ziplock bag. The unsprayed coupons just wiped with acetone stored and labelled as RW8060-58-9.
Also at 1:40 pm I coated one micro glass slide with catalyst. |
| 2:20 pm | Sprayed glass coated slide with monomer (ENB). Solvation caused to disrupt smoothness of catalyst evenly applied. |

Monomer coating solvated and pooled catalyst holes are clear, while rest is purple.



interrupt coating evenly placed on slide glass

- Subsequent spraying of monomer done to build thickness. Razor blade at edge indicates peelable coating. Allowed to dry fully before storing and labelling as RW8060-58-6 and -7 in ziplock bag.
- 3:00 pm
- Made another catalyst solution 0.75 gm in 10 ml MeCl₂.
 - We have been cleaning the flashing on each sample surface. This means that we have coated it as well.
 - After wiping surface of RW8060-58-4 sample with acetone I attempted to weigh to help determine coating weight, but couldn't get accurate reading. The 6"x6" with flashing is too large to weigh with draft shield in place and air currents in lab 5 are too great to get accurate weight. Visco tester may be used to determine thickness.

To Page No. 60

Witnessed & Understood by m ,

James B Nege

Dat

~~James B Nege~~

Invented by

R cord d by Russell Walls

Dat

~~James B Nege~~

TITLE

From Page No. _____

Catalyst Activity Study Using Tricyclohexylphosphine

Materials: Tricyclohexylphosphine (TCHP, 97% from Aldrich),
ENB (CK8148-15-1), Grubbs's Catalyst (Boulder)

Procedure:

- 1) Polymerization with ENB and the Grubbs's catalyst is very fast. TCHP was added to the catalyst in order to slow down the polymerization. Different amounts of TCHP was added to the catalyst and then the ENB syringed into the mixture. Time of polymerization was then recorded. Table of results shown below:

M/I Ratio	TCHP/I Ratio	ENB (ml)	I (gms)	TCHP (gms)	Polymerization Time
5,000:1	2:1	3.0	0.0037	0.0025	1 min.
5,000:1	5:1	3.0	0.0037	0.0063	2 min.
5,000:1	10:1	3.0	0.0037	0.0125	4 min.
5,000:1	Control	3.0	0.0037	—	13 sec.
10,000:1	2:1	3.0	0.0018	0.0013	1 min. 4 sec.
10,000:1	5:1	3.0	0.0018	0.0031	2 min. 15 sec.
10,000:1	10:1	3.0	0.0018	0.0063	4 min. 50 sec.
10,000:1	Control	3.0	0.0018	—	22 sec.

M = Monomer (ENB)

I = Initiator (Grubbs's Catalyst)

TCHP = Tricyclohexylphosphine

To Page No. _____

Witnessed & Understood by me,

Date

Invented by

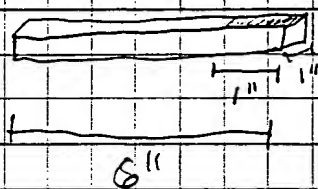
Date

Recorded by

Chris Kerk

From Page No. 50

CMF of polyolefins with
5,6-bis(chloromethyl)-2-norbornene



Polypropylene and LDPE strips were

covered, as shown in the picture on the

left, with ~ 0.1 mL each of 5,6-bis(chloro-

methyl)-2-norbornene. The area covered was 1 in^2 at the

end of a $6'' \times 1''$ strip. Two strips of each substrate

types ~~OK~~ were treated. A solution of 10 mg Grubbs' catalyst

in ~ 0.1 mL 5,6-bis(chloromethyl)-2-norbornene was

prepared and quickly applied to one of each substrate

types such that the solution was divided evenly between

the two substrates. The catalyst soln was spread

evenly over the 1 in^2 end of the substrate. The

corresponding substrate without catalyst was inverted

and placed in contact with the ~~OK~~ catalyst solution.

A 200 g weight was placed on the top of the substrates

& the reaction was allowed to cure for 24 h.

T Page No. 51

Witnessed & Understood by me,

Amy C. Burke

Date

~~OK~~

Invented by

Jate Kindell

Date

~~OK~~

Record d by

Jate Kindell

Slight initial effort, the samples peeled off with ease. That is, once a crack was formed it propagated at the adhesive-substrate interface with ease. The adhesive was a hard plastic-like material.

Amey C. Burke

Dat

Invented by

Invented by Ester K. Whall

Record d b

Record d by Forster Knudell

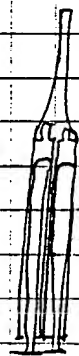
Date _____

To Page No.

From Page No. _____

Bonding of Polypropylene Using 5,6-bis(chloromethyl)-2-norbornene, $WCl_4(O-\text{C}_6\text{H}_5)_2$, + SnBuy ~~XXXX~~ delivered as a 2-part adhesive from a cartridge.

A solution of 136 mg (167 μmol) $WCl_4(O-\text{C}_6\text{H}_5)_2$ in 1 g 5,6-bis(chloromethyl)-2-norbornene + 40 mg poly ENB (synthesized in 8290-p62 sample #2) was prepared. A solution of 0.05 mL SnBuy in 1 g 5,6-bis(chloromethyl)-2-norbornene + 40 mg poly ENB (8290, p62, sample #2) was prepared. The solutions were placed in A+B sides respectively of an in-house manufactured 1 mL cartridge fashioned from 2 1 mL ~~syringes~~ syringes (plastic disposable) + a mixing chamber from the tip of a polypropylene pipette.



The mixing chamber was attached to the syringes with hot melt polyethylene adhesive.

T Pag No. _____

Witnessed & Understood by me,

Brian Canelas

Date

~~XXXXXX~~

Invented by

Peter Kuschell

Recorded by

Peter Kuschell

Date

~~XXXXXX~~

From Page N _____

The cartridge was used to deliver the adhesive to 6 sanded (w/100grit paper) polypropylene coupons over a 1 in² area. The polypropylene coupons were mated with 6 additional sanded polypropylene coupons. The joints were held in place with a 170g weight.

After 24h, 3 samples were tested. Two pulled apart by hand & a third was pulled apart on the speedy tester with a stress at break of 57psi.

After 72h, the 3 remaining samples were tested. Two pulled apart by hand & the third was pulled apart on the speedy tester with a stress at break of 217psi. The samples that pulled apart by hand were not fully cured.

Jonathan Kudall

To Page N _____

Witnessed & Understood by me.

Date

Invent d by

Date

Florian Canelas~~Florian Canelas~~

Recorded by

Jonathan Kudall~~Jonathan Kudall~~

From Pag No. —

Bonding of Polypropylene Samples using EMB + low levels of Grubbs' catalyst

A solution of 0.5 mg (0.6 μ mol) Grubbs catalyst in 1 g (8.3 mmol) 5-ethylidene-2-norbornene was prepared & quickly applied to one face of a 1" x 4" x 1/8" polypropylene substrate (sanded with 100 grit paper & washed with acetone) over a 1 in² area.

The coupons were mated with sanded polypropylene coupons such that a 1 in² overlap ~~of the~~ overlap resulted. The solution was viscous within about 30 sec & the 5th coupon pair was joined after some viscosity had built up. The bonds were held in place with a 170g weight. Monomer: Catalyst ratio = 14000:1
Samples 8327-66-1, 2, 3, 4, 5.

The procedure ~~was~~ above was repeated with 5 additional coupon pairs with the exception that

To Page N .

Witnessed & Understood by me,

Dorian Canelas

Dat

~~XXXXXXXXXX~~

Invented by

D. J. Knudell

Rec rded by

D. J. Knudell

Date

~~XXXXXXXXXX~~

TITLE

From Page No.

0.25 mg (0.3 μ mol) Grubbs catalyst was dissolved in 1 g (8.3 mmol) 5-ethylidene-2-norbornene resulting in a monomer:catalyst ratio of 28000:1. As before, the last coupon (8327-66-10) was bonded after some viscosity had built up. Samples prepared were numbered 8327-66-6, 7, 8, 9, 10.

~~SAMPLE~~ ~~RECEIVED~~ Samples were pulled on the Instron at 0.5 in/minute.

	stress @ max load
66-1	11.8 psi
66-2	12.7 psi
66-3	9.2 psi
66-4	fell apart
66-5	8.9 psi
ave	10.6 psi
std dev	1.9 psi

	stress @ max load
66-6	30.2 psi
66-7	89.9 psi
66-8	63.6 psi
66-9	81.4 psi psi
66-10	196.8 psi
ave	92.4 psi
std dev	62.7 psi

~~State~~ ~~Received~~

To Page No.

Witnessed & Understood by me,

Date

Invented by

Date

Record d by

From Pag No. _____

The Effect of Applying ^{40%} ENB/60% NBD to a two part Formulation. (5 + 10ml glass beads)

CONDITIONS

ENB	10.8g
NBD	25.2g
Europrene	6.35g
Cabosil	2.0g

PROCEDURE

ENB, NBD + Europrene were weighed into a glass jar and placed on a shaker for thirty minutes. 2g of Cabosil was added and shake for 5 minutes.

This was Side A of the formulation. It was transferred to a 10:1 Cartridge (The A-Side).

330mg of grubbs Catalyst was dissolved in 5g of ~~the~~ Dichloromethane (CH_2Cl_2) which was evaporated under flow of Nitrogen after 1.8g of Worthpar 100 processing oil was added. This resulted in a purple paste which was transferred to the B-Side of the Cartridge.

A 4" static gun and mixing tip and static gun was used to dispense the formulation. (cont glass beads)

OBSERVATION

fast curing seem to be a problem and the paste was too foamy. Also the mixing tip seem to be too long. Decided to do 50:50 ENB/NBD + reduce the mixing tip.

To Pag No. _____

Witnessed & Understood by me,

Date

Invented by

Date

Recorded by

From Page No.

Repeat study of EMB/NBD to a two part formulation. (50:50) 5ml glass beads

CONDITIONS

EMB 18g
NBD 18g
Eureprene 6.35g
Cabosil 2g

PROCEDURE Same as 8363-64

<u>RESULT 5ml</u>		<u>SAMPLE 10ml</u>			
<u>SAMPLE ID</u>	<u>LOAD (psi)</u>	<u>SAMPLE ID</u>	<u>LOAD (psi)</u>	<u>SAMPLE ID</u>	<u>LOAD (psi)</u>
836364A-1	79.30	836364B-1	100.9	836365A-1	103.6
" - 2	114.50	" - 2	96.2	" - 2	137.0
" - 3	74.87	" - 3	58.9	" - 3	89.4
" - 4	98.24	" - 4	113.9	" - 4	99.1
" - 5	113.90	" - 5	101.2	" - 5	99.7
	\bar{x} 96		\bar{x} 94		\bar{x} 106
	σ 19		σ 21		σ 18

OBSERVATIONS

All of the samples failed adhesively

J. J. J.

To Page No.

Witnessed & Understood by me,

Date

Invented by

Date

Recorded by

From Page No. _____

The Effects of Grubbs Catalyst with Cyclopentene/
Cyclohexene in a Two part Adhesive formulation on
Polypropylene Substrate. (10:1 Cartridge)

Formulations

A-Side	1A	2A
Norbornadiene	22.5g	22.5g
ENB	15.0g	15.0g
Blendex	7.5g	7.5g
Cabo-sil	1.0g	1.0g
Zeophores	4.0g	4.0g
B-Side	1B	2B
Grubbs Catalyst	0.385g	0.385g
Cyclopentene	4.615g	—
Cyclohexene	—	3.765g
Cab-O-Sil	—	0.10g
Blendex	—	0.75g

PROCEDURE

A-Side formulation (1A+2A) were combined and mixed by hand in a plastic cup, but the blendex did not dissolve in the monomers (NBD/ENB). Europrene[®] was substituted for Blendex, which was weighed in a bottle with the monomers. It was placed on a roller overnight; the formulation was successfully done the following day. A total of 2.4g of Cabo-sil was used in the formulation.

The B-Sides were done separately.

OBSERVATION

The Adhesive was found to be very fast cure, less than 15 seconds for 1B and less than 10 seconds for 2B. The 4" mixing tip had to be cut in half. We were able to get 6 samples for 1B and only 1 sample for 2B.

To Page No. _____

Witnessed & Understood by m ,

Shawn McGowan

Date

~~_____~~

Invented by

Recorded by

Jonathan Taylor

Date

~~_____~~

Proj ct No. 8374

B k No.

TITLE RESULTS

From Page No. _____

SAMPLE ID	LOAD (psi)	FAILURE MODE
83741B-1	165.68	Coh/Adh
" - 2	298.84	
" - 3	230.16	
" - 4	268.36	
" - 5	243.84	
" - 6	187.00	

$\bar{x} = 232$
 $\sigma = 45$

SAMPLE ID	LOAD (psi)	FAILURE MODE
8374112B-1	123.38	Coh/Adh
" π - 2		
" π - 3		

J. Janyon

To Page No. _____

Witnessed & Understood by me,

[Signature]

Date

[Signature]

Invent d by

Recorded by

Gonatan Jalya

Date

[Signature]

From Page No.

Repeat of 8374-11 using $\frac{1}{10}$ the amount of Grubbs Catalyst on the B-Side. The A-Side stays the same.

B-SIDE FORMULATION

	<u>1B</u>	<u>2B</u>	<u>A-SIDE</u>
Grubbs Catalyst	35mg 4.95g	35mg	Same as 8374-11
Cyclopentene	4.95g	—	
Cyclohexene	—	4.0g	
Cab-O-Sil	0.20g	0.20g 0.18g	
Europrene	—	0.8g	

PROCEDURE Same as 8374-11

RESULT

<u>SAMPLE ID</u>	<u>LOAD (PSI)</u>	<u>FAILURE MODE</u>
8374151B-1	—	
" - 2	4.20	Adh/coh
" - 3	9.90	
" - 4	7.90	
" - 5	5.46	
	\bar{x} 7	
	σ 2	

<u>SAMPLE ID</u>	<u>LOAD (PSI)</u>	<u>FAILURE MODE</u>
8374152B-1	—	
" - 2	10.82	Adh/coh
" - 3	6.20	
" - 4	5.18	
" - 5	4.90	
	\bar{x} 7	
	σ 2	

To Page No.

Witnessed & Understood by me,

Shane Miller

Date

[Signature]

Invented by

Record d by

Gina Ruffalo

Dat

[Signature]

From Page No. _____

The Effect of Grubbs Catalyst with Cyclopentene, Cyclohexene in a two part Adhesive formulation on polypropylene Substrate.

FORMULATIONNorbornadiene²A - SIDE

	<u>Amount</u>	<u>ACT Amount (x10)</u>
① Norbornadiene	22.5g	225g
② ENB	15.00g	150g
③ Europrene	7.5g	75g + 25g
④ Ca-bo-sil	1.0g	10g
⑤ Zeopheres	4.0g	40g

B - SIDE

	<u>1B</u>	<u>2B</u>	<u>1B</u>
Grubbs Catalyst	75mg	75mg	75mg
Cyclopentene	4.615g	—	2.308g
Cyclohexene	—	3.765g	2.308g
Cabo-sil	—	0.10g	0.10g
Europrene	—	0.75g	—

PROCEDURE Same as 8374-11OBSERVATION

Grubbs⁵ Catalyst and Cyclopentene cured in ~ 2 min resulting in a hard material. 50:50 Cyclopentene/Cyclohexene used instead.

To Page No. _____

With ss d & Understood by me,

Shane Mill

Date

~~Shane Mill~~

Inv nted by

Recorded by

Jonathan Taylor

Date

~~Shane Mill~~

From Page No.

SAMPLE ID	LOAD (psi)	FAILURE MODE
837422B-1	12.42	Adh / Coh
" - 2	6.64	
" - 3	10.82	
" - 4	12.62	
" - 5	—	
	\bar{x} 11	
	σ 2	

SAMPLE ID	LOAD (psi)	FAILURE MODE
837422B-1	19.60	Adh / Coh
" - 2	5.20	
" - 3	11.62	
" - 4	9.10	
" - 5	—	
	\bar{x} 11	
	σ 5	

J. Taylor
~~8/13/35~~

To Page No.

Witnessed & Understood by me,

Shane Webb

Date

~~8/13/35~~

Invent d by

Recorded by

J. Taylor

Date

~~8/13/35~~

From Page N

The Effect of Grubbs Catalyst with Cyclohexene in a two part adhesive formulation on polypropylene substrate.

A-Side
Used 8374-22 (Masterbatch)

B-SIDE

Compound	Amount
Grubbs Catalyst	200mg
Cyclohexene	3.765g
Carbo-sil	0.20g
Europrene	0.75g

PROCEDURE

The Cyclohexene and Europrene were weighed into a small vial and placed on the paint shaker for 30 minutes. The mixture was added to the Grubbs Catalyst in a plastic cup, the Carbo-sil was added next and mixed well by hand before transferring to the Cartridge. Five samples were made and cured overnight in the hood.

RESULT

SAMPLE ID	TIME	LOAD (psi)	FAILURE MODE
8374-51-1	16hr	101.86	Adh
" - 2	"	56.64	"
" - 3	"	141.98	"
" - 4	"	180.00	"
" - 5	"	312.60	Adh / Coh
		\bar{x} 159	
		σ 87	

To Page No.

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Shane ML

Date

10/1/87

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10/1/87

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SAMPLE ID	TIME	LOAD (psi)	FAILURE MODE
837451B-1	15 min	163.52	Adh
" - 2	"	152.52	"
" - 3	"	160.24	"
" - 4	"	220.04	"
" - 5	"	169.58	"
		\bar{X} 173	
		σ 24	

SAMPLE ID	TIME	LOAD (psi)	FAILURE MODE
837451C-1	30 min	193.08	Adh
" - 2	"	206.36	"
" - 3	"	217.22	"
" - 4	"	212.58	"
" - 5	"		
		\bar{X} 207	
		σ 9	

SAMPLE ID	TIME	LOAD (psi)	FAILURE MODE
837451D-1	1 hr	230.84	Adh
" - 2	"	232.62	"
" - 3	"	257.92	"
" - 4	"	278.76	"
" - 5	"		
		\bar{X} 250	
		σ 20	

SAMPLE ID	TIME	LOAD (psi)	FAILURE MODE
837451E-1	2 hr	197.52	Adh
" - 2	"	212.92	"
" - 3	"	238.80	"
" - 4	"	207.46	"
" - 5	"		
		\bar{X} 214	
		σ 15	

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From Page No. 52

The Effect of Grubbs Catalyst with Cyclohexene in a two part adhesive formulation on polypropylene substrate.

A-SIDE

Compound	Amount
80% Cyclohexyl methacrylate 20% Europrene	
Cyclohexyl Methacrylate	18.24g
Europrene	4.56g
mono-2-(Methacryloyloxyethyl) Succinate	1.45g

B-SIDE

Compound	Amount
Grubbs Catalyst	200 mg
NaEtOBET ₃	1.29g (used 1.25g)
Ca-bo-sil	0.20g
Cyclohexene	3.76g
Europrene	0.50g

PROCEDURE

The components were added together separately in a small plastic cup, hand mixed for ~ 2-5 minutes and then transferred to the cartridge. The Europrene and monomers were ^{weighed} placed into a small vial and placed on the paint shaker for 30 minutes. A static gun and a 1/4" mixing tip was used to dispense the adhesive on 20 polypropylene samples.

OBSERVATION

The samples were left to cure overnight. As they were still not cured after 2 hrs. they were pulled on the speed tester after allowing to sit for 1 week.

Result Next page

To Page No. 54

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From Pag N

RESULT

SAMPLE ID

LOAD (psi)

FAILURE MODE

837453-1	95.32	Col
" - 2	117.10	
" - 3	118.20	
" - 4	148.46	
" - 5	90.90	
" - 6	139.44	
" - 7	141.48	
" - 8	149.30	
" - 9	146.98	
" - 10	153.98	
" - 11	154.26	
" - 12	157.98	
" - 13	133.08	
" - 14	168.00	
" - 15	177.44	
" - 16	146.14	
" - 17	153.58	
" - 18	142.82	
" - 19	141.24	

 \bar{X} 140
 σ 23

J. Taylor
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From Page No. _____

7-14-00

The effect of Grubbs Catalyst with Cyclohexene in a two part adhesive formulation on polypropylene Substrate.

A-SIDE

Used 8374-22 (masterbatch)

B-SIDE

Compound	Amount
Grubbs Catalyst	200 mg
Cyclohexene	3.765g
Ca-bo-sil	0.20g
Euro prene	0.75g

PROCEDURE

Same as in 8374-51

RESULT

SAMPLE ID	TIME	LOAD (psi)	FAILURE MODE
837455A-1	5min	277.62	Adh / Coh
- 2		304.52	"
- 3		264.64	"
- 4		382.14	"
- 5		297.40	"
		\bar{x} 305	
		σ	

} samples were pulled between 5-10 minutes

SAMPLE ID	TIME	LOAD (psi)	FAILURE MODE
837455B-1	10 min	147.90	Adh
- 2		198.68	"
- 3		497.56	Coh
- 4		225.66	Adh
- 5		270.40	"
		\bar{x} 279	
		σ 120	

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From Page No. _____

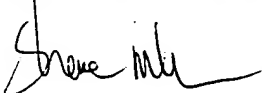
Repeated 8374.50 at 5 minutes.

SAMPLE ID	TIMES	LOAD (psi)	FAILURE MODE
8374.50C-1	5 min	112.3	Adh
" - 2	"	315.2	Adh/Col
" - 3	"	113.4	Adh
" - 4	"	151.2	"
" - 5	"	362.1	Adh/Col
		\bar{X} 211	
		σ 106	

J. Taylor
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Date

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From Page No.

The effect of Grubbs Catalyst with Cyclopentene.

A-SIDE

Used 8374-22 (masterbatch)

B-SIDE

Compound

Amount

A B

Grubbs Catalyst

200mg

140mg

Cyclopentene

4.8g

4.86g

procedure

Same as in 8374-51

RESULT

SAMPLE ID Time LOAD(psi) FAILURE MODE

837459A-1 1hr 81.50

Adh

" - 2 " 79.50

"

" - 3 " 98.34

"

" - 4 " 120.54

"

" - 5 " 63.66

"

\bar{x} 89

σ 19

Samples were pulled at 1hr-1:15

SAMPLE ID Time LOAD(psi) FAILURE MODE

837459B-1 1hr 13.00

Adh

" - 2 " 18.20

"

" - 3 " 73.62

"

" - 4 " 72.14

"

" - 5 " 38.92

"

\bar{x} 43

σ 26

To Pag No.

With ssed & Understood by me,

Shane M

Date

8/2/83

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Jonathan Taylor

Date

8/2/83

From Page N .

The Effect of Grubbs Catalyst with Cyclohexene on B-Side and ENB on A-Side. ~~8374/60~~

FORMULATIONSA-SIDE

Compound	Amount
ENB	37.5g
Europrene	7.5g
Zeospheres	4.0g
Ca-bo-sil	1.0g

B-SIDE

Compound	Amount
Grubbs Catalyst	200mg
Cyclohexene	3.765g
Ca-bo-sil	0.2g
Europrene	0.75g

A-Side formulation was prepared by adding the ENB and Europrene together in a 4oz glass jar and placed on the paint shaker; then the fillers were added. The same was done on the B-Side.

RESULT

SAMPLE ID	TIME	LOAD (psi)	FAILURE MODE
837460-1	11/15/15	81.08	Adh
" - 2	"	57.04	"
" - 3	"	82.54	"
" - 4	"	87.10	"
" - 5	"	53.52	"

 \bar{x} 72 σ 14

J. Taylor
~~8374~~

To Page No. _____

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Shane M.

Date

~~11/15/15~~

Invented by

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Jonathan Taylor

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~~11/15/15~~

From Page No.

The Effect of ^{Super} Grubbs Catalyst with Cyclohexene in a two part adhesive formulation on polypropylene substrate.

A-Side
Used Pomp Masterbatch 8374-22

Compound	Amount	B-Side's
	8374-89A	8374-89B
^{Super} Grubbs Catalyst	100 mg	50 mg
Cyclohexene	3.75 g	3.8 g
Ca-bo-Sil	0.20 g	0.20 g

* AII = 11, 20 + 30 minutes study

PROCEDURE

Both B-Sides were mixed separately by hand in a small plastic beaker and then transferred to the cartridge. Five samples were made for each set. They were allowed to cure over the week end.

Time study was done with 8374-89A.

Result

Sample ID	Time (min)	Load (psi)	Failure Mode	Comment
837489A 1	"	16.20	Adh	
" AII 2	"	392.14	Coh	Completely Cured in 25 min
" - 3	"	466.66	Adh/Coh	" "
" - 4	"	11.30	Adh	
" - 5	"	12.82	"	
		\bar{x} 180		
		σ 205		

J. Taylor

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Date

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SAMPLE ID	TIME	LOAD (psi)	FAILURE MADE	COMMENT
837489A-1	20min	15.74	Adh	
" - 2	"	11.92	"	
" - 3	"	446.24	Adh / Coh	Completely Cured in < 5min
" - 4	"	454.40	"	"
" - 5	"	474.24	"	"

SAMPLE ID	TIME	LOAD (psi)	FAILURE MADE	COMMENT
837489A-1	30min	385.96	Adh / Coh	Completely Cured in < 5min
" - 2	"	465.88	"	"
" - 3	"	465.10	"	"
" - 4	"	422.02	"	"
" - 5	"	\bar{X} 432		
		σ 31		

SAMPLE ID	TIME (hr)	LOAD (psi)	FAILURE MADE	COMMENT
837489A-1	48+	43.38	Adh	
" - 2		54.86	"	
" - 3		229.62	Coh / Adh	Completely Cured in < 5min
" - 4		41.40	Adh	
" - 5		42.70	"	
		\bar{X} 82		
		σ 74		

SAMPLE ID	TIME (hr)	LOAD (psi)	FAILURE	COMMENT
837489B-1	48+	—	—	
" - 2		18.26	Adh	
" - 3		35.92	"	
" - 4		28.02	"	
" - 5		46.68	"	
		\bar{X} 32		
		σ 10		

To Page No. ____

Witnessed & Understood by me,

Shane M.

Date

~~8/10/10~~

Invented by

Record d by

Gona Dean Taylor

Date

~~8/10/10~~